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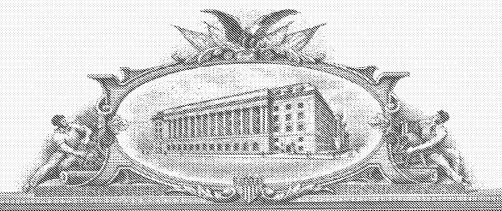
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UTILITY PATENT APPLICATION **TRANSMITTAL**

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Express Mail Label No.	EL964256034US		
Title	Devices, Materials And Methods For Sorting, Separating and Sizing Very Small Particles		
First Inventor	Neal Kalechofsky		
Attorney Docket No.	760575.00017		

See MPEP o	APPLICATION ELEMENTS chapter 600 concerning utility patent application contents.	ADDRESS TO: Commissions P.O. Box 145	A 22313-1450 OO I				
2. Submit Applica See 37 3. Specific (preferred - Description - Description - Description - Description - Cross - Stateren - Referred - Backg - Brief I - Detail - Claim - Abstraction - Abstraction - Abstraction - December - Decembe	ed arrangement set forth below) iptive title of the invention Reference to Related Applications ment Regarding Fed sponsored R & D ence to sequence listing, a table, omputer program listing appendix ground of the Invention Summary of the Invention Description of the Drawings (if filed) ed Description (s) act of the Disclosure ang(s) (35 U.S.C. 113) [Total Sheets 4]	Computer Program (Append 8. Nucleotide and/or Amino Acid Se (if applicable, all necessary) a. Computer Readable Fo b. Specification Sequence i. CD-ROM or CD-iii. Paper c. Statements verifying is ACCOMPANYING APP 9. Assignment Papers (cover 10. 37 CFR 3.73(b) Statement (when there is an assignee English Translation Docum 12. Information Disclosure Statement (IDS)/PTO-1449 Preliminary Amendment 14. Return Receipt Postcard (in Should be specifically iter 15. Certified Copy of Priority Is 16. Nonpublication Request un 16. Nonpublication Request un 17. Statement (Information Disclosure) 18. Return Receipt Postcard (in Should be specifically iter 19. Certified Copy of Priority Is Certified Copy of Priority Is Certified Return Receipt Postcard (in Should be Specifically iter 19. Certified Copy of Priority Is Celaime Nonpublication Request un 19. Certified Copy of Priority Is Celaime Nonpublication Request un 19. Certified Copy of Priority Is Celaime Nonpublication Request un 19. Certified Copy of Priority Is Celaime Nonpublication Request un 19. Certified Copy of Priority Is Celaime Nonpublication Request un 19. Certified Copy of Priority Is Celaime Nonpublication Request un 19. Certified Copy of Priority Is Celaime Nonpublication Request un 19. Certified Copy of Priority Is Celaime Nonpublication Request un 19. Certified Copy of Priority Is Celaime Nonpublication Request un 19. Certified Copy of Priority Is Celaime 19. Celaime Copy of Priority Is Celaime 19.	Computer Program (Appendix) 8. Nucleotide and/or Amino Acid Sequence Submission (if applicable, all necessary) a. Computer Readable Form (CRF) b. Specification Sequence Listing on: i. CD-ROM or CD-R (2 copies); or ii. Paper c. Statements verifying identity of above copies ACCOMPANYING APPLICATION PARTS 9. Assignment Papers (cover sheet & document(s)) 10. 37 CFR 3.73(b) Statement Power of (when there is an assignee) Attorney 11. English Translation Document (if applicable) Information Disclosure Copies of IDS Statement (IDS)/PTO-1449 Citations 13. Preliminary Amendment 14. Return Receipt Postcard (MPEP 503) (Should be specifically itemized) 15. Certified Copy of Priority Document(s) (if foreign priority is claimed) 16. Nonpublication Request under 35 U.S.C. 122 (b)(2)(B)(i). Applicant must attach form PTO/SB/35 or its equivalent.				
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DEVICES, MATERIALS AND METHODS FOR SORTING, SEPARATING AND SIZING VERY SMALL PARTICLES

Background of the Invention

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Very small particles are used in a wide variety of manufactured products. In many of these products, the particles must be of extremely small size, but the small particles must be as uniform in size as possible. Examples of products containing extremely small particles of uniform size include pharmaceuticals, abrasives, inks, high-performance liquid chromatography columns, foodstuffs, and many others.

Separation of particles by size is a critical step in the production of such particles. Particles having an average size of about 40 microns or larger can be collected by using micromachined filters. To collect, or harvest, particles having an average size smaller than about 40 microns is often accomplished by air classification. Air classification uses Stokes drag to separate particles by size. A particle falling under the influence of gravity (either the earth's gravity or an artificially induced gravitational field such as that provided by a centrifuge) in a viscous fluid medium such as air will have a terminal velocity that is strongly dependent upon the diameter of the falling particle. Differently sized particles fall through air or other viscous fluid media at different rates, thus separating in space and enabling them to be easily harvested by size.

Air classification processes works tolerably well for the separation and harvesting of particle sizes down to about two microns. However, the efficiency of the classification process depends crucially on the properties of the viscous fluid medium through which the particles are sedimenting.

But when the particles to be separated, sized, and harvested have a geometric average diameter of less than about two microns, air classification and other systems are difficult to use. Reasons for this can be inferred from figure 1:

- 1. Terminal velocities for these extremely small particles become very low.
- 2. Brownian motion begins to dominate particle dynamics.
- Particle agglomeration due to Van der Waal's attraction between particles begins to retard particle separation.

Objects of the Invention

It is accordingly the general object of this invention to provide a device for separating extremely small particles according to their average diameter or size. A related object is to provide such a device which will operate in a relatively rapid and reliable matter.

Another related object of the invention is to utilize a sedimentation medium which will encourage and permit particles of extremely small size to separate and sediment relatively rapidly and in a reliably predictable manner.

Yet another object of the invention is to provide a device for separating extremely small particles which can be operated relatively easily and at relatively small expense.

Although the preferred embodiment described below provides a device for separating particles by size, it is clear that in general the process could be extended to separate particles by shape, mass, density mechanical defect, or any other characteristic which causes some of the particles to fall through a medium faster than other particles.

Other objects and advantages of the invention will become apparent upon reading the following detailed description and upon reference to the drawings. Throughout the drawings, like reference numerals refer to like parts.

Brief Description of the Drawings

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Figure 1 is a graph showing the relationship of the geometric average diameter of particles falling through a fluid medium of known viscosity, the terminal velocity of those particles, and the Brownian displacement to which those particles are susceptible.

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Figure 2 is a phase diagram of helium.

Figure 3a is a sketch showing an extremely small particle and the wetting action imposed by a fluid surrounding the particle.

Figure 3b is a sketch similar to figure 3a showing two extremely small particles insulated from one another and deterred from agglomeration by layers of adhered atoms of the medium in which they are immersed.

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Figure 4 is a schematic drawing showing, in sectional aspect, the top of a device for separating, sizing and classifying extremely small particles.

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Figure 5 is a schematic drawing showing, in sectional aspect, the bottom of the device shown in figure 4 for separating, sizing and classifying extremely small particles.

Figure 6 is an image derived from a scanning electronic microscope showing the top layer of a mixture of 7 micron and 2 micron diameter particles prior to sedimentation in superfluid helium.

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Figure 7 is an image derived from a scanning electron microscope showing the top layer of a mixture of 7 micron and 2 micron diameter particles after sedimentation in superfluid helium.

20 <u>Detailed Description</u>

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While the invention will be described in connection with certain preferred embodiments and procedures, it will be understood that it is not intended to limit the invention to these embodiments or procedures. On the contrary, it is intended to cover all alternatives, modifications and equivalents as may be included within the spirit and scope of the invention as defined by the appended claims.

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To accomplish the above objects, the invention comprises a quantity of low viscosity high wetting parameter fluid; means for injecting the particles to be sorted, separated and sized into that fluid; and means for harvesting at least some of those separated particles from the fluid.

The properties of superfluid helium make it an excellent medium in which to separate small particles. Ordinary liquid helium at 4.2 degrees Kelvin has a viscosity 5.5 times

less than that of air at 20 degrees centigrade. A low viscosity medium suggests a relatively high terminal velocity for particles passing through the medium. In addition, if a low temperature can be maintained in the medium, the effect of Brownian diffusion on particle dynamics will be minimized. Furthermore, liquid helium has a very high wetting parameter; that is, helium atoms have a greater affinity for foreign objects than they do for other helium atoms. As a result, and as suggested in figures 3a and 3b, solid particles 10 immersed in liquid helium 12 quickly become insulated from one another in layers 14 of adhered helium atoms that have only a very weak Van der Waals attraction to one another. The layers of helium atoms thus acts as a surfactant to deter particle agglomeration while the particles are immersed in the cold liquid. Accordingly, liquid helium is a good candidate for a better particle sedimentation protocol than protocols currently achievable using air classifying equipment or other currently available techniques.

However, because liquid helium will boil under ordinary conditions, superfluid helium is an even better sedimentation medium. Superfluid helium can be produced relatively simply by lowering the pressure above a container filled with ordinary liquid helium. The physical properties of superfluid helium are so different from ordinary helium liquid helium, or Liquid Helium I, that superfluid helium is considered to be a unique state of matter; it is neither a solid nor liquid nor gas. Either 3He or 4He or a combination of 3He and 4He can be used.

The pressure and temperature constraints for superfluid helium or Liquid Helium II are shown in the phase diagram of figure 2. To generalize somewhat, superfluid helium can be produced and maintained at pressures less than 2.5 atmospheres and temperatures below 2 degrees Kelvin. In accordance with the invention, superfluid helium can be used to efficiently, effectively and inexpensively separate and sort extremely small particles.

A device for sorting, separating and sizing extremely small particles is suggested schematically in figures 4 and 5. To separate extremely small particles according to their average diameter or size, and to do so in a relatively rapid, reliable yet inexpensive manner in accordance with the invention, the illustrated device comprises, a cryogenic chamber 40 within which particle movement and separation

can occur; a loading chamber 20 connected to the cryogenic chamber 40 for loading particles into the cryogenic chamber, and a collector device 80 connected to the cryogenic chamber 40 for collecting at least some of the particles after they have been separated by size.

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The closed, gas-tight loading chamber 20 includes a receiver 22 for receiving particles 10, 11 and 13 to be separated and for loading the particles 10, 11 and 13 into the cryogenic chamber 40. A gate valve 23 is interposed between the receiver 22 and the cryogenic chamber 40 for controlling the flow of particles from the loading chamber 20 to the cryogenic chamber 40.

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Above or upstream of the receiver 22, a vacuum conduit 25 is connected via appropriate valving 27, 28 to a vacuum or exhaust pump (not shown) for drawing air from the receiver 22. A delivery conduit 29 delivers helium to the receiver 20 when appropriate valving 30, 27 is opened. The particles 10, 11, 14 to be classified, sorted and sized can be delivered from a remote source (not shown) through a conduit 32 and inlet valve 34 to the receiver 22. At appropriate time, the gate valve 23 is opened and the particles flow from the receiver 22 through a delivery conduit 36 extending into the interior of the cryogenic chamber 40 by a sufficient distance so that the particles are deposited within superfluid in the cryogenic chamber 40.

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In accordance with one aspect of the invention, the cryogenic chamber is adapted to produce and maintain a column of very low viscosity, high wetting fluid such as superfluid helium 4He. An OptistatSXM Helium bath cryostat can be adapted and used for this purpose. This device is available from Oxford Instruments

Superconductivity USA of 130A Baker Avenue Extension, Concord, Massachusetts.

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As indicated above, particles falling through the superfluid medium in the cryogenic chamber tend to separate according to their size; larger particles tend to fall faster and arrive at the bottom of the column before the slower-falling smaller particles. To distinguish between these differently sized particles in accordance with another aspect of the invention, differentiation or size recognition equipment 90 can be provided, as suggested in figure 5. In the illustrated embodiment, this particle size indicating and recognition equipment 90 takes the form of a laser 91 mounted to direct a beam of

light 92 through windows 93 and 94 in the cryogenic chamber. Light which illuminates the particles falls on a target screen 95. The laser should provide light at a frequency far from that absorbed by the superfluid so that the heat load on the superfluid helium is minimized. For example, a Nd:YAG laser operating on a low duty cycle at the 532 nm line may be effective. As the particles fall through the laser light beam, diffraction patterns are created on the receiving screen 95. Differently sized particles create differing diffraction patterns. Differences in the diffraction patterns can be detected and sensed by a computer 96 connected to the target screen 95, and information about the particles sizes can be delivered to the system operator by any suitable means.

This information about particles sizes can be used to harvest particles of a desired size or sizes and to discard particles which are excessively large or excessively small. This particle harvesting can be accomplished in any of a number of ways. For example, particles 11 which are too large will reach the bottom of the chamber apparatus first, before any particles of the desired size arrive. Under the circumstances, the superfluid helium in a discard conduit 42 can be pumped out, drawing off the oversized particles 11 with the fluid. Thereafter, when particles of the desired size begin to reach the bottom of the column, discard column pumping is halted and the superfluid helium and right-sized particles can be drawn-off from the column 40 by a harvest conduit 44 and pump (not shown). When particles 13 which are too small to meet requirements begin to arrive at the bottom of the column 40, pumping and particle draw off or removal through the harvest column 44 can be halted and particle withdrawal through the discard column 42 can be resumed.

Alternatively, a diverter baffle 47 can be located at the column bottom as illustrated in figure 5, and the diverter baffle 47 can be connected by a shaft or any other suitable means 48 to a baffle control 49 as illustrated in figures 4 and 5. The diverter baffle is oriented, sized and located to direct particles falling upon it to either a discard portion 48 of the column bottom or to a harvest or collection portion 49 of the column bottom. The operation of this diverter baffle can be controlled by the particle size sensing computer 97.

Claims:

- 1. A device for separating very small particles by size, comprising, in combination,
- a cryogenic chamber within which particle travel and separation can occur,
 a loading chamber connected to the cryogenic chamber for loading particles into the
 cryogenic chamber, and
 a collector device connected to the cryogenic chamber for collecting at least some of the
 - a collector device connected to the cryogenic chamber for collecting at least some of the particles after they have been separated by size.
- 10 2. A device according to claim 1 further including a device for indicating the size of the particles as the particles move through the cryogenic chamber.
 - 3. A device according to claim 1 wherein said cryogenic chamber is adapted to produce and maintain a column of very low viscosity, high wetting fluid.
 - 4. A device according to claim 1 further including a superfluid within said cryogenic chamber.
 - 5. A device according to claim 4 wherein said superfluid is a liquid.
 - 6. A device according to claim 5 wherein said liquid is helium.
 - 7. A device according to claim 6 wherein said helium is 4He.
- 8. A device according to claim 1 wherein said cryogenic chamber is adapted to produce an internal temperature of lower than 2.2 degrees Kelvin.
 - 9. A device according to claim 1 further including a discard conduit for drawing off particles to be discarded.
 - 10. A device according to claim 1 further including a harvest conduit for drawing off particles to be harvested.
- 25 11. A device according to claim 1 further including a discard conduit for drawing off particles to be discarded, a harvest conduit for drawing off particles to be harvested, and an actuator for actuating a flow of fluid in the discard conduit and a flow of fluid in the harvest conduit at different times.
- 12. A device according to claim 1 wherein said loading chamber includes a receiver for
 30 receiving the particles to be separated and for loading the particles into said cryogenic chamber.
 - 13. A device according to claim 12 further including a gate valve interposed between said receiver and said cryogenic chamber for controlling the flow of particles from the loading chamber to the cryogenic chamber.

- 14. A device according to claim 12 wherein said loading chamber further includes a vacuum conduit for drawing air from the receiver.
- 15. A device according to claim 12 wherein said loading chamber further includes a delivery conduit for delivering helium to the receiver.
- 5 16. A device according to claim 13 further including a delivery conduit extending from the receiver into the cryogenic chamber by a sufficient distance that the particles flowing from the receiver and the gate valve are deposited within superfluid in the cryogenic chamber.
- 17. A device according to claim 1 wherein said collector device includes a diverter for diverting a flow of particles to be discarded from a flow of particles to be harvested.
 - 18. A device according to claim 1 further including a laser generator positioned to direct a beam of laser light through the cryogenic chamber so as to illuminate any particles therein.
 - 19. A device according to claim 18 further including a detection device for detecting a diffraction pattern of light created by said illumination of particles within the cryogenic chamber.

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- 20. A device according to claim 19 further including recognition apparatus for detecting and distinguishing between various diffraction patterns of light created by said illumination of particles within the cryogenic chamber, and for generating different signals in response to the detection of different diffraction patterns.
- 20 21. A device according to claim 20 further including means connecting the recognition apparatus to a diverter mechanism for diverting a flow of particles of a predetermined sizes from a flow of particles of other sizes.
 - 22. A method of sorting very small particles by size, comprising the steps of providing a column of very low viscosity high wetting fluid,
- 25 introducing a plurality of particles of various sizes into the fluid in the column, causing the particles to travel through the fluid so as to separate the particles by size, and harvesting the separated particles of desired size.
 - 23. A method according to claim 21 including the steps of causing the particles to travel through the fluid toward at least one collecting point, and harvesting at least some particles at the collecting point.
 - 24. A method according to claim 22 wherein the step of providing a column of very low viscosity high wetting fluid includes the step of providing a superfluid.
 - 25. A method according to claim 22 wherein the step of providing a superfluid includes the step of providing a helium superfluid.

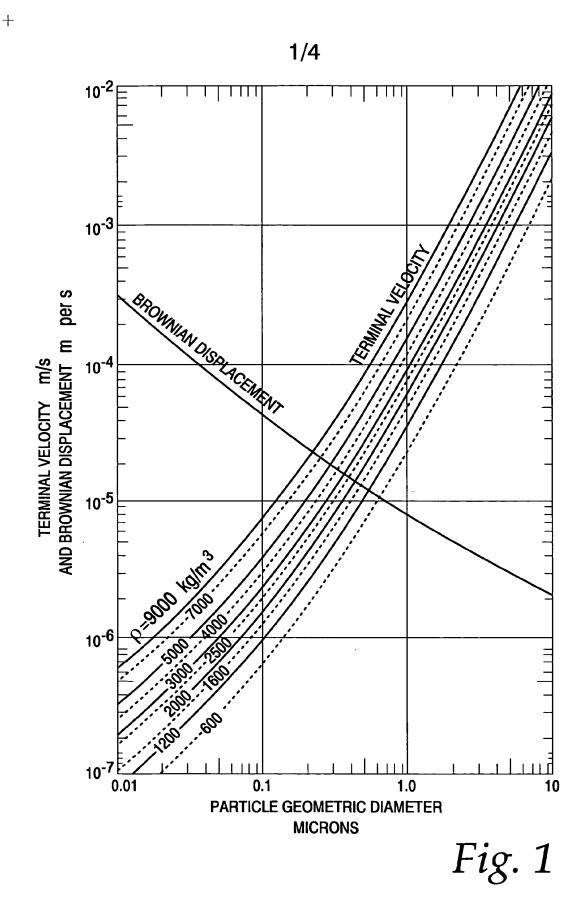
- 26. A method according to claim 22 further including the steps of illuminating the particles traveling through the fluid, creating diffraction patterns by the illumination of the particles, and distinguishing between particles of different sizes by differences in said diffraction patterns.
- 5 27. The method according to claim 22 further including the steps of loading particles into a loading chamber receiver and drawing air from the loading chamber receiver before introducing the particles into the cryogenic chamber.
 - 28. The method according to claim 27 further including the steps of introducing gaseous helium into the loading chamber receiver after at least some air is drawn from the loading chamber receiver.
 - 29. A method according to claim 22 further including the step of drawing particles to be harvested from the cryogenic chamber.
 - 30. A method according to claim 22 further including the step of drawing particles to be discarded from the cryogenic chamber.
- 15 31. A method according to claim 22 further including the steps of drawing particles to be harvested from the cryogenic chamber at a predetermined time, and drawing particles to be discarded from the cryogenic chamber at a different time.
 - 32. A batch of particles of predetermined sizes which have been selected by the method of claim 31.
- 20 33. A batch of particles wetted by helium.
 - 34. A superfluid having therein a plurality of very small particles.
 - 35. A combination according to claim 34 wherein at least some of said particles are less than 40 microns in size.
 - 36. A combination according to claim 34 wherein at least some of said particles are less
- 25 than 2 microns in size.

- 37. A combination according to claim 33 wherein said helium is in a superfluid state...
- 38. A combination according to claim 33 wherein said helium has a temperature of less than 2.2 degrees Kelvin.
- 39. A combination according to claim 34 wherein said superfluid is contained in a30 vertically elongated chamber.
 - 40. The use of a superfluid helium to separate and sort particles.
 - 41. The use according to claim 40 wherein said superfluity is helium.
 - 42. The use of helium to separate and sort particles.

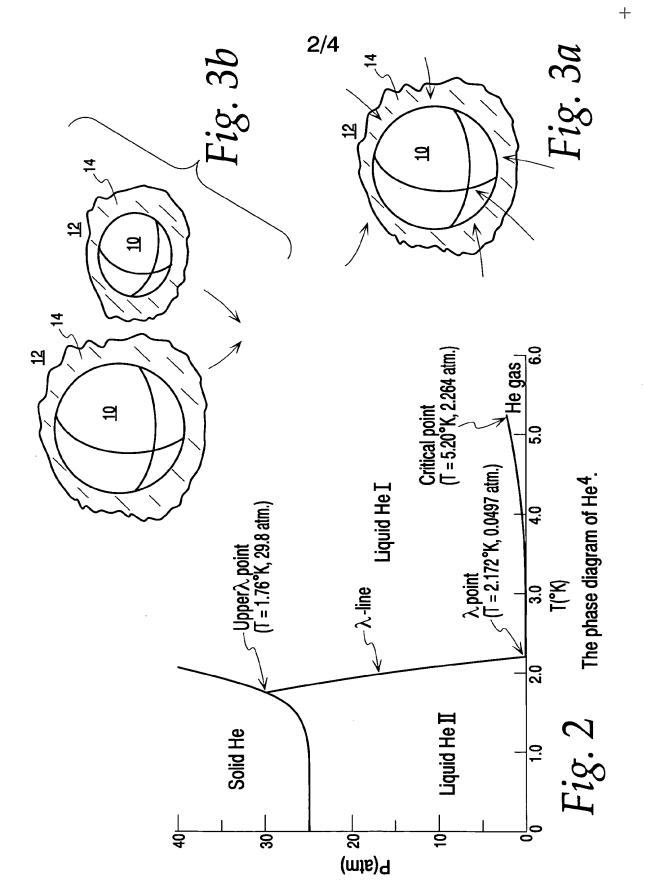
Abstract of the Invention

Advice for sorting separating and sizing very small particles is disclosed and claimed. The device comprises a cryogenic chamber within which particle movement, travel and separation can occur; a particle loading chamber for loading particles into the cryogenic chamber; and a particle collector. Also disclosed and claimed is helium, and more specifically helium in its superfluid state, for separating the particles.

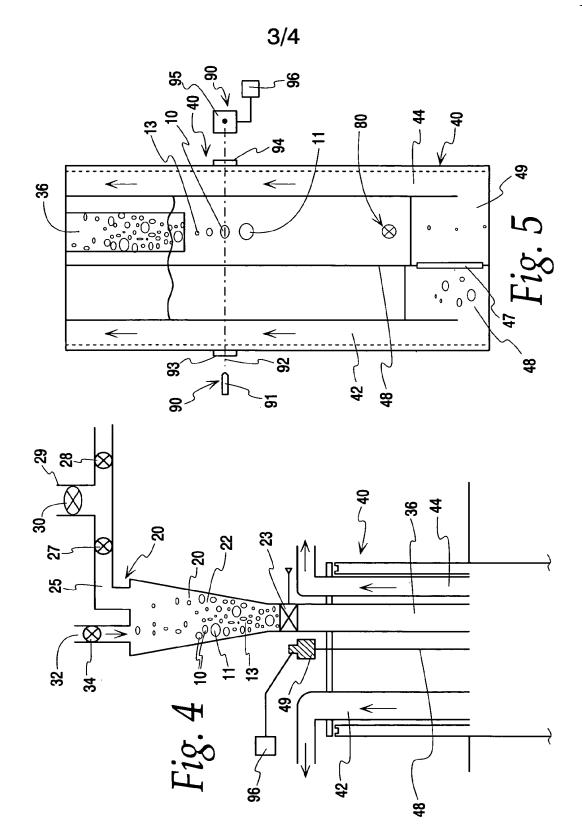
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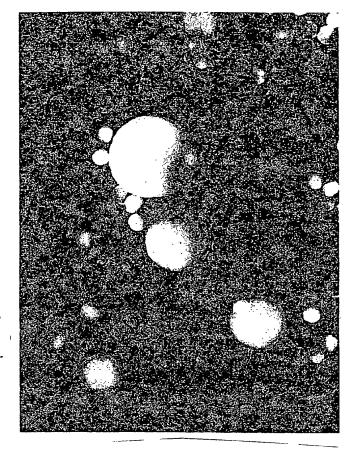


Fig. 7

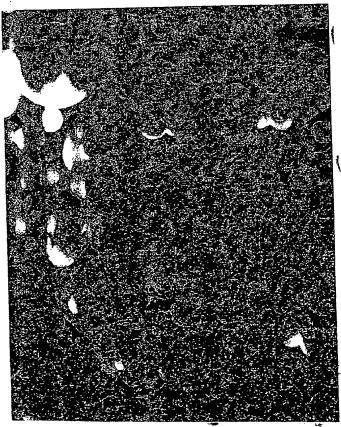


Fig. 6

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DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a design patent is sought on the invention entitled:

"DEVICES, MATERIALS AND METHODS FOR SORTING, SEPARATING AND SIZING VERY SMALL PARTICLES"

Case No. 760575.00	0017, the specification of which	
	x is attached hereto.	
(check one)	Domestic Priority from U.S. Provisional Patent Application. X was filed on October 28, 2003 as Application Serial No. 60/514,949 and was amended on (if applicable)	- -

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims as amended by any amendment referred to above.

I acknowledge the duty to disclose to the United States Patent Office all information which is known to me to be material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, 1.56.¹

I do not know and do not believe this invention was ever known or used in the United States of America before my or our invention thereof, or patented or described in any printed publication in any country before my or our invention thereof or more than one year prior to this application, that the same was not in public use or on sale in the United States of America more than one year prior to this application, and I believe that the invention has not been patented or made the subject of an inventor's certificate issued before the date of this application in any country foreign to the United States of America on an application filed by me or my legal representatives or assigns more than twelve months prior to this application, and that no application for patent or inventor's certificate on this invention has been filed in any country foreign to the United States of America prior to this application by me or my legal representatives or assigns:

And I hereby appoint Todd S. Parkhurst (26,494), Lewis T. Steadman, Sr. (17,074) and Robert J. Depke (37,607), all members of the firm of Holland & Knight LLC
Telephone: (312) 263-3600

as my attorney with full power of substitution and revocation, to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith and direct that all correspondence be forwarded to:

Holland & Knight LLP

⁽b) Under this section, information is material to patentability when it is not cumulative to information already of record or being made of record in the application, and

⁽¹⁾ It establishes, by itself or in combination with other information, a prima facie case of unpatentability of a claim; or

⁽²⁾ It refutes, or is inconsistent with, a position the applicant takes in:

⁽i) Opposing an argument of unpatentability relied on by the Office, or

⁽ii) Asserting an argument of patentability.

A prima facie case of unpatentability is established when the information compels a conclusion that a claim is unpatentable under the preponderance of evidence, burden-of-proof standard, giving each term in the claim its broadest reasonable construction consistent with the specification, and before any consideration is given to evidence which may be submitted in an attempt to establish a contrary conclusion of patentability.

131 S. Dearborn, 30th Floor Chicago, Illinois 60603

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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